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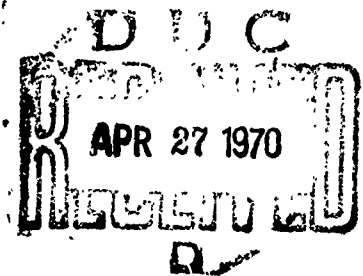
AFRPL-TR-69-219

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**(U) THE SYNTHESIS OF NEW IONIC
INTERHALOGEN OXIDIZERS**

F. Q. ROBERTO

OCTOBER 1969



TECHNICAL REPORT AFRPL-TR-69-219

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THE SYNTHESIS OF NEW IONIC
INTERHALOGEN OXIDIZERS (U)

Francisco Q. Roberto

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FOREWORD

(U) This is a phase report of work conducted at the Air Force Rocket Propulsion Laboratory to determine the feasibility of producing interhalogen high-energy liquid oxidizers. This work was accomplished under Project 314801ACL during the period of July 1968 through July 1969 by Dr. Francisco Q. Roberto, Task Scientist. The author wishes to acknowledge the valuable technical discussions and support furnished by Dr. Claude Merrill, Project Scientist; Dr. Charles Bock for X-ray diffraction work and to Mr. John H. Leahy for the analysis of this hazardous and very reactive material. The author also wishes to acknowledge Dr. George Begun, Oak Ridge National Laboratory for running the Raman spectral study.

(U) This report has been reviewed and approved.

NORMAN J. VANDER HYDE
Chief, Solid Propellant Branch
Propellant Division
Air Force Rocket Propulsion Laboratory

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CONFIDENTIAL ABSTRACT

(C) The synthesis of hexafluorochloronium (VII) hexafluoroplatinate (V), $\text{ClF}_6^+\text{PtF}_6^-$, from the reaction of chlorine pentafluoride and platinum hexafluoride is described as an oxidation-reduction reaction. This has been confirmed by elemental analyses, infrared and Raman Spectroscopy, X-ray powder diffraction and displacement reaction with trifluorochlorine oxide. This is the first example of a perfluorinated heptavalent chlorine cation. The compound, $\text{ClF}_6^+\text{PtF}_6^-$, has been indexed as having a cubic structure based on preliminary X-ray powder pattern.

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SECTION I INTRODUCTION

(U) Reactions of platinum hexafluoride with other oxidizing agents have been studied by several investigators.⁽¹⁻⁶⁾ With chlorine trifluoride, difluorochloronium (III) hexafluoroplatinate (V) (ClF_2PtF_6), and chlorine pentafluoride⁽⁷⁾ are produced.⁽⁶⁾ Gortsema and Toeniskoetter⁽⁶⁾ also attempted to react platinum hexafluoride with chlorine pentafluoride; the only product observed, in addition to the reactant, was a yellow solid believed to be ClF_2PtF_6 (it was assumed that it resulted from the decomposition of chlorine pentafluoride into chlorine trifluoride and subsequent reaction of chlorine trifluoride with platinum hexafluoride). Recent studies of the same reaction have shown that tetrafluorochloronium (V) hexafluoroplatinate (V) was produced.⁽⁸⁾ Other salts of tetrafluorochloronium ion have been reported recently.⁽⁹⁾

(C) Studies in this laboratory of platinum hexafluoride with chlorine pentafluoride have shown that in an excess of chlorine pentafluoride using a sapphire reactor, a solid product, believed to be hexafluorochloronium (VII) hexafluoroplatinate (V) (ClF_6PtF_6), was obtained. The cation ClF_6^+ has been reported previously.^(10, 11) However, most of the evidence was based on infrared data and more recent studies have disputed its synthesis.⁽¹²⁾ The solid produced in this laboratory is markedly different from those reported⁽¹⁰⁻¹²⁾ and new data are presented for the synthesis of ClF_6^+ in this laboratory.

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SECTION II EXPERIMENTAL PROCEDURES

A. EQUIPMENT

(U) The vacuum system used in this work consisted of a nickel manifold constructed from 1/4-inch nickel pipe to which a number of subsystems were attached. These were: (1) a multipurpose line for handling, measuring, and transferring reactants; (2) a line for purifying and storing fluorine; and (3) a similar line for purifying halogen fluorides. All valves (Hoke TM 413 monel diaphragm valves with metal seats) were silver-soldered to the manifold outlets. Pressure measurements were made with the Wallace and Tiernan gage, Model FA 145, accurate to 0.2 torr. Volumes were calibrated using pure helium and nickel bulbs of known volume.

(U) The vacuum system and sapphire reactors were passivated with fluorine at 300° using a heat gun. Passivation was considered complete when pressure at ambient temperature was identical before and after heating to 300°. Before each run, the whole system was passivated with chlorine pentafluoride followed by a final passivation with platinum hexafluoride.

B. MATERIALS

(U) Platinum hexafluoride was obtained from Ozark-Mahoning Company, Tulsa, Oklahoma. The material was further purified by low-temperature distillation according to the procedure described.⁽¹⁾

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The infrared spectrum⁽¹³⁾ and vapor pressure of the pure material agreed well with the published values.

(C) Chlorine pentafluoride and trifluorochlorine oxide, ClF_3O ,⁽¹²⁾ were obtained from Rocketdyne, a Division of North American Aviation, Canoga Park, California. The material was used without further purification.

C. ANALYSIS

(U) The platinum compounds were transferred into a 50-ml quartz reactor that had been previously weighed in the dry box and purged with dry nitrogen. The reactor was then connected to the vacuum system to remove the nitrogen and reweighed. The sample was frozen at -196° and 5-10 ml of double-distilled water was transferred to the sample. Immediate reaction was accompanied by a flash when vapor initially came into contact with the sample. The sample was warmed to ambient temperature by removing the liquid nitrogen trap. After half an hour at ambient temperature, the solution was refrozen to -196° and 5-10 ml of hydrazine was transferred to it. The solution was again warmed to ambient temperature. The platinum solids formed by hydrolysis and reduction were filtered off; they were redissolved with aqua regia to convert the platinum-containing materials to chloroplatinic acid. The solution was diluted with water, reduced with sodium formate,⁽¹⁴⁾ and the finely divided platinum metal was filtered, dried, and weighed. After additional dilution, aliquots of the stock solution were analyzed for chlorine and fluorine. Chloride ions were determined by anodic chronopotentiometry using a silver electrode.

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Fluoride ion was determined potentiometrically with an Orion fluoride ion electrode.⁽¹⁵⁾ The above methods were checked with standard samples.

(U) X-ray diffraction studies were carried out using the powder techniques with samples sealed in 0.3-mm thin-wall quartz capillary tubes. The tubes were previously flamed in a vacuum line to remove moisture. Samples were loaded in a dry box, flushed with dry nitrogen, sealed with Kel-F grease, and placed in a 5.73-cm Debye-Sherrer camera. Measurements were made using copper $K\alpha_1$ radiation with a nickel filter. Exposure times varied from 1/2 hour to 4 hours. Intensities were estimated visually.

(U) Infrared spectra were obtained with Beckman IR-5A and Perkin-Elmer 337 spectrophotometers. Gaseous spectra were obtained using a 10-cm nickel cell equipped with silver chloride windows. Spectra were obtained on the solid pressed between chloride windows of the nickel infrared cell. All operations were carried out in a dry box.

D. REACTION OF PLATINUM HEXAFLUORIDE WITH CHLORINE PENTAFLUORIDE

(C) In a typical reaction, 1.26 mmole (0.390 grams) of platinum hexafluoride, and 3.78 mmole (0.491 grams) of chlorine pentafluoride were condensed at -196° into an evacuated, prepassivated 7-cc sapphire reactor. The reaction mixture was slowly warmed to ambient temperature. The mixture was left exposed to light at $22-23^\circ$ for a period of 8 days. The reaction was monitored by the disappearance of PtF_6 (red gas). As time progressed, the red-brown solid phase became lighter. After 8 days,

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the solid turned to the bright yellow color of the ClF_xPtF_6 salt.

The reactor was cooled to -196°C and found to contain no noncondensable gas. The gaseous mixture was found to contain only ClF_5 .⁽¹⁶⁾ The excess ClF_5 was removed under vacuum giving a bright orange-yellow solid with no vapor pressure at room temperature. The weight of the solid product was 0.489 grams. The material was analyzed according to the procedure described.

(C) Anal. Calcd. for ClF_6PtF_6 : Pt, 42.56; F, 49.7; Cl, 7.73.

Found: Pt, 42.7; F, 47.2; Cl, 5.2.

(C) The infrared spectrum of the yellow solid was prepared in the dry box by pressing it between silver chloride windows. This is shown in Figure 1. The X-ray powder pattern of the solid is given in Table I.

E. REACTION WITH ClF_3O

(C) In order to further characterize the ClF_xPtF_6 , excess ClF_3O was condensed on top of the solid of ClF_xPtF_6 at -196°C . The reactor was gradually warmed to -85°C using trike-liquid nitrogen slush. There was very little evidence of reaction at this temperature for 30 minutes. At -75°C , there was some reaction and the only volatile product found was ClF_5 and a trace of ClFO_2 . This reaction continued up to -35°C producing more ClF_5 . The total noncondensable (-196°C) was 5 mm after about 2 1/2 hours. The reaction was left overnight starting at -35°C and gradually warming to room temperature. The 40 millimeters of noncondensable at -196°C was identified (by mass spectral analysis and reaction

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with mercury) as fluorine. The ClF_5 formed was transferred and weighed by pumping on the reactor at -85°C . After all the volatile product was removed, the solid was weighed and subjected to infrared analyses. The infrared spectra was identical to the spectra of $\text{ClOF}_2\text{PtF}_6$ ⁽¹⁷⁾ previously obtained between the reactions of ClF_3O and PtF_6 .

(7) Based on the weight of fluorine, chlorine pentafluoride and $\text{ClF}_2\text{OPtF}_6$ produced, the ClF_xPtF_6 was calculated to contain a 50:50 mixture of ClF_4PtF_6 and ClF_6PtF_6 within experimental error. These results definitely established the presence of ClF_6^+ in the reaction product. The reaction to produce the heptavalent ClF_6^+ can be written as:



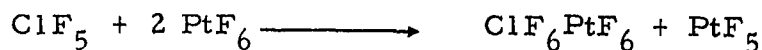
In excess ClF_5 , PtF_5 reacts with it to give ClF_4PtF_6 .

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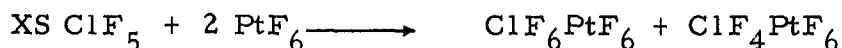
SECTION III

RESULTS AND DISCUSSION

(C) The solid product obtained in the reaction between platinum hexafluoride and chlorine pentafluoride using a sapphire reactor activated with light is believed to be ClF_6PtF_6 , in accordance with the equation

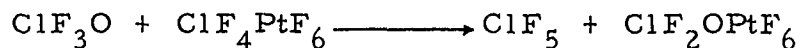


In excess ClF_5 , the reaction proceeds further to give ClF_4PtF_6 . The overall reaction in excess ClF_5 can be expressed as:



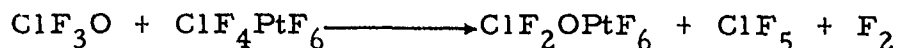
The reaction can be described as an oxidation-reduction where chlorine is oxidized to the heptavalent state forming an ionic solid $\text{ClF}_6^+\text{PtF}_6^-$ and PtF_5 . However, in an excess ClF_5 , PtF_5 is acting as a Lewis acid abstracting a fluoride ion from ClF_5 to give ClF_4PtF_6 .

(C) Further evidence for the synthesis of ClF_6PtF_6 was obtained from the reaction with trifluorochlorine oxide. The mass balance obtained from the reaction of $\text{ClF}_4\text{PtF}_6 \cdot \text{ClF}_6\text{PtF}_6$ with ClF_3O indicates that ClF_4PtF_6 reacted with ClF_3O at -75° to -35° according to the equation:

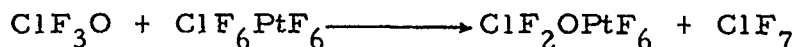


Above -35° , melting point of ClF_3O , ClF_6PtF_6 then reacted with ClF_3O in one of two ways:

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or



Since the only volatile products observed at ambient temperature were ClF_5 and fluorine, it is implied that ClF_7 formed and is unstable at this temperature or it is catalytically decomposed by $\text{ClF}_2\text{OPtF}_6$. From this study the composition of the mixture was deduced to be a 50:50 mixture of ClF_6PtF_6 and ClF_4PtF_6 based on the weight of fluorine, ClF_5 and $\text{ClF}_2\text{OPtF}_6$ produced from the above equation.

(C) The infrared spectrum of solid mixture $\text{ClF}_6\text{PtF}_6 + \text{ClF}_4\text{PtF}_6$ pressed between silver chloride plates consists of bands attributed to ClF_4PtF_6 and bands at 889, 875 and 540 cm^{-1} assigned to ClF_6^+ and the peak at 649 cm^{-1} attributed to the PtF_6^- absorption band. The 890, 875 and 540 cm^{-1} observed in the infrared were absent in the Raman spectrum as expected of an octahedral molecule. Since ClF_6^+ is isoelectronic with the octahedral, SF_6 , some similarity in their spectra is expected, except for slight frequency shifts due to their mass difference. The higher mass of the central atom in ClF_6^+ compared to SF_6 should cause a shift toward lower frequencies, as is indeed observed for the infrared active bands.

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Going from SF_6 (940 cm^{-1} , γ_3) to ClF_6^+ (890 cm^{-1} , γ_3), the shift is 50 cm^{-1} ; γ_4 for SF_6 at 615 cm^{-1} is shifted by 65 cm^{-1} in ClF_6^+ (540 cm^{-1}). These assignments are tentative and require further refinements.

(C) Of the six normal modes of vibration expected of an octahedral ion of the type XY_6 , ($A_{1g} + E_g + 2 F_{1u} + F_{2g} + F_{2u}$), only two of these modes ($2 F_{1u}$) will be infrared active and three will be Raman active (A_{1g} , E_g , and F_{2g}).

(C) The remaining F_{2u} mode is inactive in both infrared and Raman spectrum. However, since the octahedral PtF_6^- mode of vibrations have not been assigned, it is rather difficult to assign vibrations due to ClF_6^+ with any certainty. Since the only absorption band that we can assign with any degree of certainty to PtF_6^- is the absorption band at 645 cm^{-1} , the other absorption bands, in addition to 890, 875 and 540, at 521 and 313 cm^{-1} , may be attributed to ClF_6^+ .

(C) The X-ray powder pattern for ClF_6PtF_6 (after subtracting the lines due to ClF_4PtF_6) is relatively simple, indicating high symmetry. The high symmetry of the unit cell of ClF_6PtF_6 (probably cubic) seems reasonable since both ClF_6^+ and PtF_6^- have octahedral symmetries. The only other known compound of the $\text{XF}_6^+ \text{YF}_6^-$ structure is $\text{IF}_6^+ \text{SbF}_6^-$.⁽¹⁸⁾ This salt has a high symmetry (face-centered cubic) and ClF_6PtF_6 is expected to exhibit similar structural symmetry.

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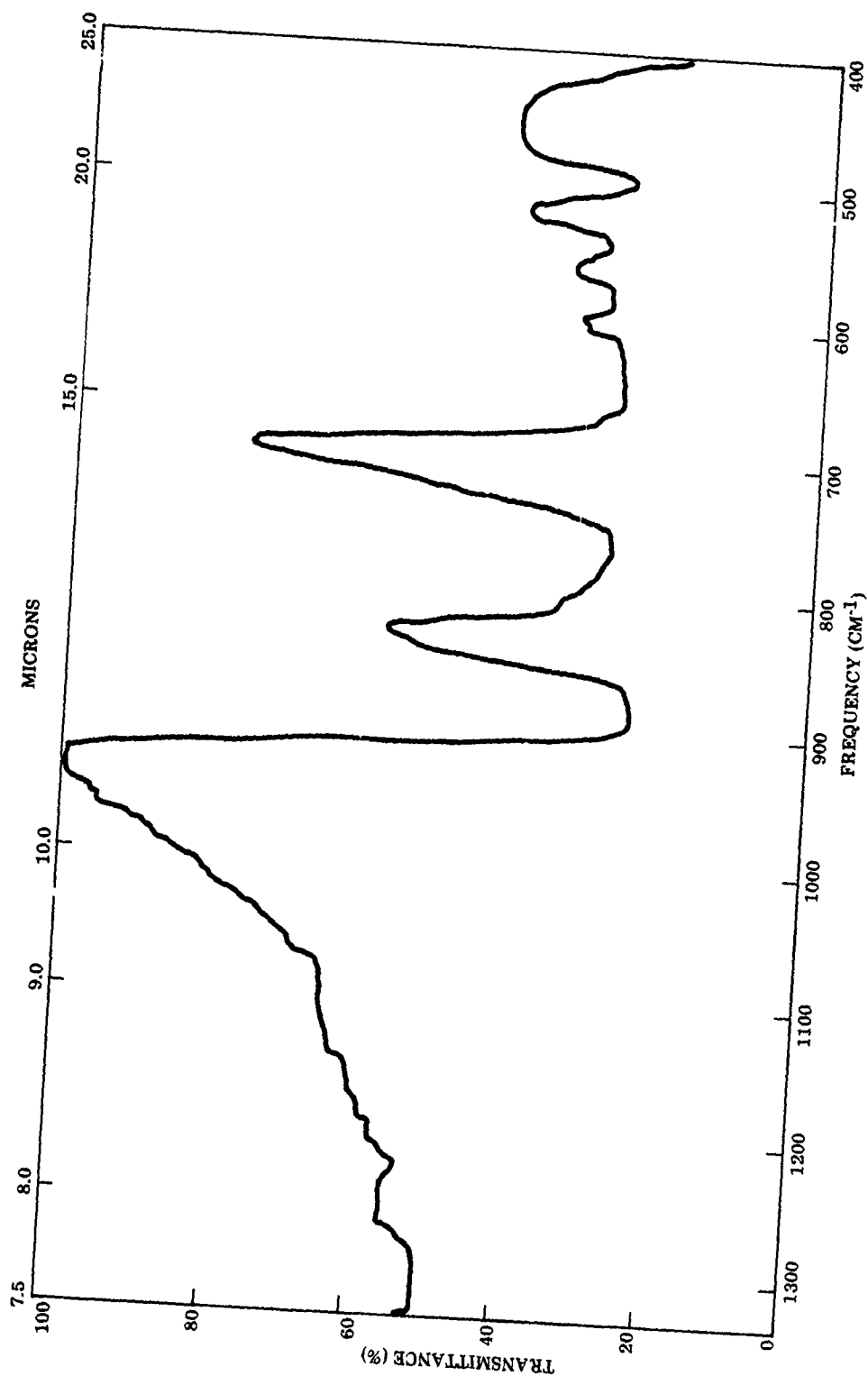


Figure 1. Infrared Spectrum of the Mixture of $\text{ClF}_6^+ \text{PtF}_6^-$ and $\text{ClF}_4^+ \text{PtF}_6^-$

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TABLE I. (C) X-RAY POWDER DATA FOR $\text{ClF}_6^+ \text{PtF}_6^-$ *

$d_{(\text{obsd})}$	$d_{(\text{calcd})}$	hkl	Intensity
5.717	5.74	110	Strong
4.036	4.06	200	Very Strong
3.328	3.32	211	Medium
2.576	2.569	310	Light
2.334	2.345	222	Medium
1.999	2.030	400	Light
1.923	1.915	411	Very Light
1.774	1.772	421	Very Light
1.727	1.732	332	Very Light
1.530	1.535	520, 432	Very Light

* Tentatively identified as having a cubic symmetry.

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13. ABSTRACT (C) The synthesis of hexafluorochloronium (VII) hexafluoroplatinate (V), $\text{ClF}_6^+\text{PtF}_6^-$, from the reaction of chlorine pentafluoride and platinum hexafluoride is described as an oxidation-reduction reaction. This has been confirmed by elemental analyses, infrared and Raman spectroscopy, X-ray powder diffraction and displacement reaction with trifluorochlorine oxide. This is the first example of a perfluorinated heptavalent chlorine cation. The compound, $\text{ClF}_6^+\text{PtF}_6^-$, based on preliminary X-ray powder pattern, has been indexed as having a cubic structure.		

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